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(54) Title of the Invention:Skin Ointment

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# Transcription of Specifications (No amendment to content) SPECIFICATION

## 1. Title of the Invention Skin ointment

#### 2. Claims

A skin ointment which consists of a combination of 4-(1,1-dimethylethyl)-4'-methoxydibenzoylmethane and organic acids and/or their salts.

## 3. Detailed Description of the Invention

[Field of Industrial Application]

The present invention relates to a skin ointment with exceptional effects concerning the prevention of discolouration and the prevention of a reduction in ultraviolet absorptivity, and which consists of a combination of 4-(1,1-dimethylethyl)-4-methoxydibenzoylmethane and organic acids and/or their salts.

4-(1,1-dimethylethyl)-4'-methoxydibenzoylmethane has recently drawn attention as a skin ointment component for ultraviolet absorbers such as sunscreens and foundations which aim to protect against ultraviolet radiation.

[Prior Art]

However, the sole use of 4-(1,1-dimethylethyl)-4'-methoxydibenzoylmethane in a skin ointment results in colouration and discolouration over time, resulting in evident damage to the appearance of the product and a fall in the quality of the same as a skin ointment. According to the inventors, this has also been found to bring about a drop in the ultraviolet absorptivity of 4-(1,1-dimethylethyl)-4'- methoxydibenzoylmethane, resulting in a reduction in the

protective effect against ultraviolet radiation. Conventionally, although toners were added in advance to skin ointments to conceal the colouration and discolouration induced by the 4-(1,1-dimethylethyl)-4'-methoxydibenzoylmethane, this failed to provide a fundamental solution to the problem. Furthermore, there was no method of approach at all concerning the fall in ultraviolet absorptivity.

[Problems to be Solved by the Invention]

Given the above circumstances, the inventors, as a result of diligent research, discovered that a skin ointment could be obtained which provided a simultaneous solution to the above problems by combining the 4-(1,1-dimethylethyl)-4'-methoxydi benzoylmethane with organic acids and/or their salts.

[Means for Solving the Problems]

The present invention relates to a skin ointment with minimal deterioration in product quality due to the combination of 4-(1,1-dimethylethyl)-4-methoxydibenzoyl methane and organic acids and/or their salts.

The configuration of the present invention is described below.

The 4-(1,1-dimethylethyl)-4`-methoxydibenzoylmethane as used in the present invention constitutes a compound with the following structure

and is manufactured using a method as shown in Kokai number (1980)66535. Furthermore, this product is currently marketed by Givaudan under the trade name 'Parsol 1789'.

Although loads for the skin ointment are random, loads for those in system-soluble form should preferably be below 20% by weight.

Known organic acids and/or their salts which are used in the present invention consist of glyconic acids, ascorbic acid, succinic acid, citric acid, lactic acid, tartaric acid, butyric acid, oxalic acid, malonic acid, valeric acid, formic acid, acetic acid and propionic acid, where more than 1 or 2 acids are used.

Loads should be 1/5000-20 times the 4-(1,1-dimethylethyl)-4'-methoxydibenzoyl methane by weight, or preferably 1/1000-10 times by weight. A

load less than 1/5000 times by weight would result in a dilute concentration of the organic acids and/or their salts, which would be insufficient as a solution to the above problem. On the other hand, a load greater than 20 times by weight would not alter the results, and would simply be costly and uneconomical.

Skin ointments which can combine 4-(1,1-dimethylethyl)-4'-methoxydibenzoylmethane and organic acids and/or their salts may also consist of any standard skin ointment base such as solubilisation bases, emulsification bases, powder bases, powder dispersion bases, double-layered cosmetics with water/oil/powder bases.

Skin ointments of the present invention may contain natural extracts such as animal oils and ester oils, triglycerides, higher fatty acids and higher alcohols, polyalcohols and other sugar derivatives, humectants such as pyrolidone carboxylic acid, thickeners such as water-soluble polymer compounds and clay minerals, preservatives, surfactants, sequestrants, ultraviolet absorbers and organic or inorganic powders, pigments, medical ingredients, dyestuffs and perfumes as required, providing the quantity or quality of the above does not result in a loss of effect of the present invention.

#### [Effects of the Invention]

Skin ointments of the present invention have the following advantages. The addition of 4-(1,1-dimethylethyl)-4 -methoxydibenzoylmethane to the product prevents colouration and discolouration over time and enables the maintenance of product quality by preventing the reduction of ultraviolet absorptivity.

#### [Examples]

Next, a detailed description of the present invention is provided based on test examples and examples. Furthermore, the present invention is in no way limited to these examples.

#### Test Example 1

The colouration and discolouration over time in sunscreen with the following formulation was observed, where the content of 4-(1,1-dimethylethyl)-4-methoxydibenzoylmethane was 5% by weight and where the content of organic acids and/or their salts was 0% by weight, 0.005% by weight, 0.01% by weight, 0.05% by weight, 0.1% by weight and 0.5% by weight respectively.

Cetanol	0.5%	
Vaseline	0.070	2.0
Squalane	7.0	2.0
Self-emulsifiable glyceryl monostearate	7.0	
	2.5	
Polyoxyethylene sorbitan ester monostearate		
(20E.O.)	1.5	
4-(1,1-dimethylethyl)-4'- metoxydibenzoylmetha	ne	5.0
Jojoba oil	5.0	0.0
B.	0.0	
Propylene glycol	5.0	
Glycerin	0.0	5.0
Beegum (montmorillonite)	5.0	3.0
Potassium hydroxide	0.3	
Organic acid and/or their salts		
Water	0-0.5	
-Method of Manufacture-	balance	:

A (oil phase) and B (aqueous phase) were heated respectively at 70 until completely dissolved. A was added to B, which was then emulsified using an emulsifier. The emulsified product was cooled using a heat exchanger to obtain the sunscreen.

Colouration was evaluated immediately after emulsification, and discolouration over time was evaluated after storage for 1 month at  $50\Box$ .

Results are shown in Tables 1-3.

Table 1 (with Ascorbic Acid)

Ascorbic Acid Load	Colouration (immediately after emulsification)	Discolouration (after 1 month)
0%	х	x
0.005	0	
0.01	. 0	0
0.05	0	0
0.1	0	0
0.5	0	0

Colouration (discolouration) present	X	
Slight colouration (discolouration)		
Colouration (discolouration) absent	0	

Table 2 (with Sodium Citrate)

Sodium Citrate Load	Colouration (immediately after emulsification)	Discolouration (after 1 month)
0%	X	x
0.005		
0.01	0	0
0.05	0	0
0.1	0	0
0.5	0	0

(below as blank)

Table 3 (with Glyconic Acids)

Glyconic Acid Load	Colouration (immediately after emulsification)	Discolouration (after 1 month)
0%	х	x
0.005	· 0	0
0.01	· 0	0
0.05	. 0	0
0.1	0	0
0.5	0	0

The results of Tables 1-3 show that the skin ointments of the present invention are stable, quality products which show neither colouration immediately after manufacture or discolouration over time.

#### (below as blank)

Test Example 2

The sample used in Test Example 1 was applied to a quartz glass plate at a thickness of 5 microns, and the ultraviolet absorption spectrum was measured using a spectrophotometer.

Tables 4-6 show data concerning the reduction rate of the absorption peaks for samples kept for 1 month at 50□ in relation to the absorption peaks immediately after preparation of the sample.

Table 4 (with Ascorbic Acid)

Ascorbic Acid Load	Reduction Rate of Absorption Peak (%)
0%	22
0.005	15
0.01	9
0.05	5
0.1	3
0.5	1

Table 5 (with Sodium Citrate)

Sodium Citrate Load	Reduction Rate of Absorption Peak (%)
0%	22
0.005	13
0.01	9
0.05	7
0.1	4
0.5	2

(below as blank)

Table 6 (with Glyconic Acids)

Table 6 (With Glycome Heras)		
Glyconic Acid Load	Reduction Rate of Absorption Peak (%)	
0	22	
0.005	11	
0.01	7	
0.05	5	
0.1	3	
0.5	11	

(below as blank)

Example 1: Cream		
A. Stearic acid	10.0%	
Stearyl alcohol	4.0	
Butyl stearate		8.0
Monoglycerin ester stearate	2.0	
4-(1,1-dimethylethyl)-4'- methoxydi benzoylmethan	e 2.0	
Perfume	0.4	

Preservative	dosage
B. Propylene glycol	10.0
Glycerin	4.0
Multitol	1.0
Potassium hydroxide	0.4
Sodium lactate	0.05
Purified water	balance

The oil phase section of A and the aqueous phase section of B were heated respectively at 70 $\square$  until completely dissolved. The A phase was added to the B phase, which was then emulsified using an emulsifier. The emulsified product was cooled using a heat exchanger to obtain the cream.

Ex	kample 2: Cream		
A.	Cetanol	4.0%	
	Vaseline	7.0	
	Isopropyl myristate	8.0	
	Squalane	15.0	
	Monoglycerin ester stearate	2.2	
	POE (20) sorbitan monostearate	2.8	
	4-(1,1-dimethylethyl)-4'- methoxydibenzoylmethane	0.5	
	Perfume	0.3	
	Antioxidant	dosage	
	Preservative	dosage	
B.	Glycerin	10.0	
	Dipropylene glycol	5.0	
	Ascorbic acid		0.01
	Purified water	balance	;

The cream was obtained in accordance with Example 1.

#### Example 3: Latex

A. Squalane	5.0%	
Oleyl olate	3.0	
Vaseline	2.0	
Sorbitan sesquioleate		0.8
Polyoxyethylene oleylether (20E.O.)	1.2	
4-(1,1-dimethylethyl)-4`- methoxydibenzoylmethane	1.5	
Perfume	0.3	
Preservative	dosage	
B. 1.3 Butylene glycol	5.0	
Ethanol	3.0	
Carboxyvinyl polymer		0.2
Potassium hydroxide	0.1	
Sodium citrate	0.05	
Purified water	balance	:

The latex was obtained in accordance with Example 1.

#### Example 4: Foundation

A. Cetanol	3.5%
Deodourising lanolin	4.0
Jojoba oil	5.0

Vaseline	2.0
Squalane	6.0
Monoglycerin ester stearate	2.5
POE (60) hardening castor oil	1.5
POE (20) cetyl ether	1.0
4-(1,1-dimethylethyl)-4`- methoxydibenzoylmethane	8.0
Preservative	dosage
Perfume	0.3
B. Propylene glycol	10.0
Balanced powder	12.0
Citric acid	0.2
Sodium citrate	0.2
Purified water	balance

The foundation was obtained in accordance with Example 1.

Example	5:	Cosmetic	Liquid
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A. Ethanol	5.0%	
POE oleyl alcohol ether	2.0	
2-ethylhexyl-P-dimethylaminobenzoate		0.18
4-(1,1-dimethylethyl)-4'- methoxydibenzoylmethane	0.02	
Perfume	0.05	
B. 1.3 Butylene glycol	10.0	
Glycerin	5.0	
Ascorbic acid		0.4
Purified water	balance	e

The cosmetic liquid was obtained by the addition of the alcohol phase of A to the aqueous phase of B and the solubilisation of the above.

Ltd

Patent Applicant: Shiseido Co.,

# Procedural Amendments (voluntary) April 16, 1985

Attn: Commissioner, Patent Office: SHIGA, Manabu

1. Case
Patent Application Number 60(1985)-56491

2. Title of the Invention Skin Ointment

3. Party making Amendment

Relation to Case:

Patent Applicant

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4. Subject of Amendment Specification, whole text

5. Content of Amendment

Transcribing of Specification (as based on offset printing, as in attached sheet (no amendment to content))